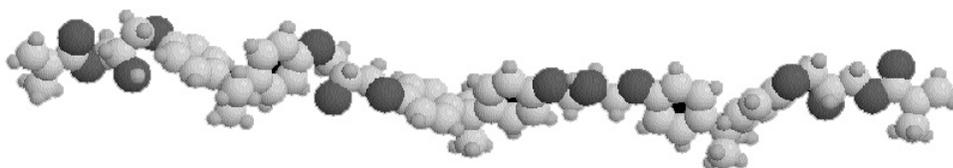

Technical Research



CoREZYN Premium Vinyl Ester Molecule

Optimizing Initiator Systems for Cured-In-Place Pipe Infrastructure Repair

INTERPLASTIC CORPORATION
Thermoset Resins Division

ABSTRACT

Using Cured-In-Place Pipe (CIPP) for infrastructure repairs continues to grow. The ability to repair a section of sewer/waste pipe in a single day without disruption of the users' service, plus its attractive economics, make this technique a leader in the infrastructure repair market. Conservative estimates show this industry growing 10 to 20 percent annually.

Optimizing the gel and cure process of the resin, which allows crews do the CIPP insertions faster, has been an industry improvement target to help maintain and advance its economic edge. Fewer man-hours required to complete a job lowers the overall cost and gives this technique an even greater economic advantage over other infrastructure repair techniques like dig and replace, pipe jacking, slip lining, deformed/reformed thermoplastic pipe, spiral wound thermoplastic, coatings and panels. This research discusses optimizing the initiator and curing temperature to shorten the required time on the job site.

A designed experiment was conducted using an isophthalic resin developed for CIPP applications. It was combined with various initiators commonly used in CIPP applications and they were combined at various levels. The study looks at the gel and cure profile from 127° to 180°F (52° to 82°C) and the stability of the initiated resin at temperatures ranging from 50° to 90°F (10° to 32°C). Models of the data were generated and used to determine the optimum initiators and their levels leading to shortened time on the site. The effects of lot-to-lot variation of the low temperature initiator were also studied to see if this would cause a change in day-to-day performance.

INTRODUCTION

Infrastructure aging is in line with the general aging of metropolitan areas across North America. As the infrastructure ages, remediation methods are developed to restore the infrastructure without undue economic hardship. The needs of the municipality and businesses along the deteriorated route must be considered.

A CIPP remediated line is expected to have the physical properties equivalent to or in excess of the host pipe's. The service life of the remediated line is expected to be a minimum of fifty years if the liner is properly installed and cured.

The time required to complete an installation is an important factor for the CIPP market. Developing optimum initiator systems that reduce time on a job site is a continual area of research for the suppliers to this market. The reduction in site time also helps the CIPP market share expand and become more cost competitive with other pipe relining/replacement technologies.

Several initiator systems are used in the CIPP market. The commonly used systems consist of a low temperature and a high temperature peroxide. There are some single initiator systems like cumene hydroperoxide, but they are used in "over-the-hole" insertions where they can utilize a much shorter catalyzed pot life (as short as 10 hours compared to other conventional initiator systems with a 24 to 48 hour pot life). The initiator systems are selected based on recommendations from the resin/initiator suppliers. The recommendations are derived from factors such as liner thickness, diameter and length; ground water conditions; desired catalyzed stability; type of insertion; equipment; and the availability of initiators.

The focus of this work is on the most commonly used low temperature peroxide initiator, 1-Di-(4-t-butylcyclohexyl)-peroxydicarbonate. This peroxide is currently sold as Akzo Nobel® Perkadox 16, Norac® Norox 600, Aztec® BCHPC and Crompton Espercarb® 1043.® We evaluated three commonly used levels of this peroxide in combination with three levels of four commonly used high temperature peroxides: tertiary butyl perbenzoate; tertiary butyl peroxy-2-ethylhexanoate; tertiary butyl peroxy-3,5,5-trimethyl hexanoate; and the mixture consisting of 25% 1,1-Di-(butylperoxy)-3,3,5-trimethylcyclohexanoate; 25% dibutyl phthalate and 50% tertiary butyl peroxy-2-ethylhexanoate.

EXPERIMENTAL

A designed experiment using Design Expert® by Stat Ease evaluates cure performance temperatures ranging from 121° to 180°F (49° to 82°C). The designed experiment also analyzes their stability at temperatures ranging from 50° to 90°F (10° to 32°C) for four different initiator systems.

All of the work was done with a single lot of a 1:1 isophthalic/maleic, all propylene glycol, unsaturated, polyester polymer commonly used in the corrosion industry.^{1, 2} This was done to eliminate variations caused by differences in the polymer solution. This polymer was converted into COR72-AT-470HT, which is a blend of the unsaturated polyester resin, styrene,

alumina trihydrate and other additives to enhance performance. Interplastic Corporation formulated this product to use with Di-(4-t-butylcyclohexyl)-peroxydicarbonate (the primary initiator) and it is commercially available to the CIPP market.

Samples of resin with initiator were prepared according to the following procedure: The solid initiator, Di-(4-t-butylcyclohexyl)-peroxydicarbonate, was used in each system. It was made into a slurry with equal amounts of styrene. Different liquid initiators were used as the secondary initiators in this study. These peroxides do not require the additional step of pre-dissolving them in styrene before use. They can be mixed directly into the resin.

These liquid peroxides were: tertiary butyl perbenzoate (System I); tertiary butyl peroxy-2-ethylhexanoate (System II); tertiary butyl peroxy-3,5,5-trimethyl hexanoate (System III); and the mixture consisting of 25% 1,1-Di-(butylperoxy)-3,3,5-trimethylcyclohexanoate; 25% dibutyl phthalate and 50% tertiary butyl peroxy-2-ethylhexanoate (System IV).

The desired amounts of initiators were weighed in a glass container and then 800 grams of resin was added and mixed for 30 to 45 seconds. The mixture was allowed to rest at $77^{\circ} \pm 2^{\circ}\text{F}$ ($25^{\circ}\text{C} \pm 1^{\circ}\text{C}$) for 15 minutes. Then it was poured into four 4-ounce (118 ml) glass jars and four 19 x 150 mm test tubes (Kimble 45050 Soft Glass with Lip). The jars were placed in their respective incubators or baths for stability testing. The test tubes were placed in their respective elevated temperature water baths for cure studies.

The cure studies were done using a variation in the standard SPI Gel Time Test Method, Interplastic Corporation CRSTP 12. A quantity of initiated resin was prepared and a portion of it was poured into four 19 mm x 150 mm test tubes (Kimble 45050, Soft Glass with Lip) to a depth of 3 in. (76.2 mm), which is approximately 30 grams. A 1/8-inch (3.2 mm) diameter J couple thermal probe was inserted into the resin and centered from the sides of the test tube. The bottom of the probe was set at 1.5 in. (38.1 mm) from the bottom of the test tube. The test tubes were immersed in the water bath so the top of the resin was at least 0.5 in. (12.7 mm) below the surface of the water and then the strip chart recorder was turned on.

The cure data was run at 120°F (49°C), 140°F (60°C), 160°F (71°C) and 180°F (82°C). These temperatures were chosen based on the typical practices and capabilities found in the field in the CIPP industry.

This data included the gel time, which is defined as the time it takes the resin to go from 30°F (16.7°C) below bath temperature to 10°F (5.5°C) above bath temperature.

The cure time is defined as the time it takes for the resin to go from 30°F (16.7°C) below bath temperature to maximum temperature.

The gel-to-peak exotherm interval is defined as the time it takes the resin to go from 10°F (5.5°C) above bath temperature to the maximum temperature.

Peak exotherm is the maximum temperature. The elevated temperature cure testing was run in a Lindberg Blue M model number WB1110A circulatory water bath.

The method used to determine the stability was based on a 100-gram mass in a 4-oz (118-ml) jar. Exposing the resin to a light source can greatly shorten stability, so the sealed samples that were placed in the water bath were covered with aluminum foil to eliminate any exposure to light.

A temperature probe connected to a chart recorder was inserted in the resin for the samples tested at 65°F (18°C), 77°F (25°C) and 90°F (32°C).

The samples at 50°F (10°C) were visually inspected every one to two days, typically daily, by turning the container upside down and visually inspecting the bottom of the container for any signs of gelation. The stability (gel point) was defined as the first appearance of solid (gelled material) in the bottom of the container. Once any solid material was found, the number of days to reach the first formation of gelled material was recorded. The sample was monitored for another two or three days to confirm the gel point by continual growth in the size of the gelled mass.

The stability (gel point) tests were run at 65°F (18°C), 77°F (25°C) and 90°F (32°C) and monitored on the strip chart recorder. The definition of stability (gel point) was the time when the temperature of the container reached 2°F (1°C) above temperature set in the incubator/bath.

Two different sets of apparatus were used to determine the stability. The 50°F (10°C) and 65°F (18°C) tests were run in an incubator (EchoTherm™ Model IN30 by Torrey Pines Scientific Inc.) that has sub-ambient capabilities to 40°F (4°C).

The 77°F (25°C) and 90°F (32°C) work was done using a water bath (Lindberg Blue M model WB112A).

The temperature settings of the baths and incuba-

tors were calibrated using a NIST traceable thermometer. This thermometer was an ERTCO 200°C L-NIST-TT 76 mm 1 mm.

RESULTS

Gel Time

Graphing the gel time/stability data from 50° to 180°F (10° to 82°C) using a log scale for time and no transformation on the temperature scale showed a linear relationship with least squares analysis. The R^2 of each of the sets of data was over 0.99. Data is compiled in Table 5. Figure 1 contains a typical plot of the data. The gel time curves for the four different mixtures of initiators gave similar plots. The slopes were similar but there was a slight range of the intercept values.

The mixtures that only contained the lowest level of primary initiator gave the longest stability/gel time at temperatures at or below 90°F (32°C).

The higher level of primary initiator and the combinations of primary and secondary initiators gave similar stability/gel time at or below 90°F (32°C).

The gel times at temperatures over 90°F (32°C) were very similar for the all of the combinations of initiators tested.

Cure Time

The cure time data from 120° to 180°F (49° to 82°C) was graphed using a log scale for time and no transformation on the temperature scale showed a linear relationship with least squares analysis. In some cases, the peak exotherm did not meet the definition of the gel time so the cure time could not be determined. The R^2 of each set of data was over 0.99 and this can be seen in Table 6. Figure 2 contains a typical plot of the data.

The cure time curves had similar slopes for all four mixtures of initiators. Based on that observation it appears that the primary initiator had the most influence on the ultimate cure time. Samples with the low level of the primary initiator had the longest cure time, even with the presence of the secondary initiator.

The general trend of the response showed that as the level of primary initiator was increased, the cure time shortened.

The cure time showed a high dependence on the

primary initiator. The three levels evaluated appeared as three different sets of data. The lowest level corresponded with the longest cure time, followed by the next highest level and the highest level had the fastest cure. Little difference was seen in the secondary initiator at or below 160°F (71°C). A difference was seen in the mixtures with the secondary initiator at 180°F (82°C). When the secondary initiator is present, the mixture has a faster cure time than the mixtures have without it. The phenomenon was present in all four systems.

Gel-To-Peak Exotherm Interval

The data points at 120°F (49°C) did not give consistent results and are not used in this analysis. The low temperature caused the resin to gel but not cure to a high crosslinked state. This was observed in the low peak exotherm, which exceeded the temperature of the water bath by less than 15°F (8.3°C). The resin-initiator mixtures did not meet the definition of the gel time in all cases and those are noted in the tables. When the mixture did not meet the gel time definition, the gel-to-peak exotherm interval could not be determined.

Graphing the gel-to-peak time data from 140° to 180°F (60° to 82°C) using a log scale for time and no transformation on the temperature scale showed a linear relationship with least squares analysis. The R^2 of each set of data was over 0.99. Figure 3 contains a typical plot of the data.

The mixtures with only the primary initiator had similar slopes and at the higher concentration, the line shifted to lower values.

As the temperature was increased, the mixtures with the combination of primary and secondary initiators had more slope so the gel-to-peak time was faster compared to the mixtures without the secondary initiator. In three of the four cases, the mixture with the highest level of initiator (primary and secondary) had the shortest gel-to-peak time at 180°F (82°C). In the fourth case, it was the same as the mid-range points.

The gel-to-peak time begins to reach a limit of approximately 0.5 minutes as you increase the temperature of the water since the reaction cannot happen instantaneously.

The gel-to-peak exotherm interval is a better indication of the speed of cure since it does not include the induction time (time for the initiator to form sufficient free radicals to cause the resin to gel) so only the time to cure the resin is considered. This is essentially the time needed to cure the resin once it gels.

In all four systems, the low level of the primary initiator had the slowest cure time. The highest level of primary and secondary initiators had the fastest gel-to-peak exotherm interval. The other systems fell between these two mixtures. Typically at 180°F (82°C), the mixture with the highest level of primary initiator and no secondary initiator was slower than the mixtures with the various combinations of primary and secondary initiators used in this study.

Peak Exotherm

The data points at 120°F (49°C) did not give consistent results. This was due to the peak exotherms being so low and difficult to determine. In some cases, the peak exotherm did not exceed the bath temperature by more than 10°F (5.5°C). A typical graph of the peak exotherm data from 140° to 180°F (60° to 82°C) using no transformations on the temperature or time scales appeared to be fairly linear over this temperature range. It is shown in Figure 4.

The mixtures that contained only the primary initiator had lower peak exotherms than the mixtures with the combination of initiators. The mixture with the primary initiator at 0.56% had peak exotherms of 20° to 40°F (11° to 22°C) lower than the mixture with 0.94%. This was 20° to 40° F (11° to 22°C) lower than the mixtures that contained the secondary initiators.

The peak exotherm data for System I showed a 16°F (8.9°C) range of peak exotherms at 140°F (60°C), and a 6°F (3.3°C) range at 160°F (71°C) and at 180°F (82°C).

The peak exotherm data for System II showed a 20°F (11°C) range of peak exotherms at 140°F (60°C), a 20°F (11°C) range at 160°F (71°C) and an 8°F (4.4°C) range at 180°F (82°C).

The peak exotherm data for System III showed a 29°F (16.1°C) range of peak exotherms at 140°F (60°C), and a 20°F (11°C) range at 160°F (71°C) and a 12°F (6.7°C) range at 180°F (82°C).

The peak exotherm data for System IV showed a 30°F (16.7°C) range of peak exotherms at 140°F (60°C); 20°F (11°C) range at 160°F (71°C) and a 13°F (7.2°C) range at 180°F (82°C).

DISCUSSION

The primary initiator had the most affect on the low temperature stability/gel time of the mixture.

Changes in the primary initiator level significantly changed the stability/gel time at temperatures below 90°F (32°C) of the initiated mixture. Lower levels of initiator gave the longest cure. As the level of primary initiator was increased from 0.54% to 0.94%, the stability/gel time decreased by as much as 65% at 50°F (10°C).

The primary initiator also gave the lowest peak exotherm when used without a secondary initiator. Even at 0.94%, it was still significantly lower than the mixtures of primary and secondary initiators in all four systems.

The secondary initiators affected the stability/gel time, gel-to-peak interval and peak exotherm.

System III least affected the stability at low temperatures. Less than a 20% decrease was seen at 50°F (10°C) with the incorporation of 0.38% of the secondary initiator.

Systems IV and I had a 40 - 50% decrease at 50°F (10°C).

System II had the biggest drop, over 60% at 50°F (10°C).

The gel-to-peak interval appeared related to the total initiator level in the mixture - the higher the initiator level, the shorter the gel-to-peak interval. As the bath temperature increased for the gel time test, the differences between the gel-to-peak times decreased. This could be due reaching a lower test limit.

The secondary initiators all increased the peak exotherm more than comparable levels of the primary initiator. Each of the systems yielded similar peak exotherms at the different temperatures at which the gel time tests were run.

CONCLUSION

There are some critical factors to consider for CIPP applications/installations. The installers want the longest stability possible so they have a safety factor in how much time they have to get the material in the pipe. They also want a short "cooking" cycle that allows them to manufacture a quality liner in the shortest time once it's in the host pipe. The third important parameter is to get enough heat generated during the cooking step to drive the resin cure to an acceptable level but not hot enough to distort, crack or cause excessive shrinkage in the liner.

The parameters tested in this study that relate to the

stability issue are the stability/gel times at and below 90°F (32°C).

The parameter that reflects the cooking time is the gel-to-peak exotherm interval.

The heat generated during the “cooking step” is reflected in the peak exotherm.

Choosing a primary and secondary initiator combination that yielded the longest stability and the fastest gel-to-peak exotherm interval would lead to the selection of System IV. This system had the best stability/gel time at temperatures below 90°F (32°C) and the fastest gel-to-peak exotherm interval. It appears that this secondary initiator had the least affect on the stability at low temperatures but still gave a fast gel-to-peak interval of the four tested.

Systems III and I were the next best. The secondary initiator used in System II had the most deleterious affect on the low temperature stability and thus was the lowest ranked system.

The peak exotherm, especially controlling it, is important to the installation and the final appearance of the bag’s urethane liner. Too high an exotherm causes the bag’s urethane liner to blister. A high exotherm also affects the resin’s cure rate. Excessive shrinkage and cracking of the bag can occur if the cure rate is too fast or the exotherm too high.

This data gives the end-user some insight as to the selection of a mixture to control the exotherm and rate, which is critical in thick liner applications. The initiator system used in thick applications of over 1 in. (25mm), have higher levels of the primary initiator and low to no secondary initiator to minimize the system’s peak exotherm.

FOOTNOTES

1. BP plc Bulletin IP-86c.
2. BP plc Bulletin PIA 96b.

AUTHORS

David Herzog is the Director of Research and Development for the Thermoset Resins Division of Interplastic Corporation. He received a MS in chemistry from the University of Minnesota in 1983 and a BA from Carleton College in 1980.

Anthony Bennett is the Manager of the Corrosion and Specialty and Laminating Resin Groups for the Thermoset Resins Division of Interplastic Corporation.

Jay Lampert is a Resin Chemist in the Thermoset Resins Division of Interplastic Corporation.

Jason Schiro is a Senior Resin Chemist in the Thermoset Resins Division of Interplastic Corporation.

DATA

Table 1: System I - Results using Di-(4-t-butylcyclohexyl)-peroxydicarbonate as the low temperature initiator and t-butyl perbenzoate as the high temperature initiator.

Low Temp. Initiator, %		0.56	0.56	0.75	0.75	0.75	0.94	0.94
High Temp. Initiator, %		0.00	0.38	0.19	0.19	0.19	0.00	0.38
Test	Unit							
120°F (48.9°C)								
Gel Time	minutes	60.3	69.7	53.2	48.9	47.8	32.2	41.9
Cure Time	minutes	60.3	69.8	53.2	51.8	51.5	39.2	47.4
Peak Exotherm	°F	128	131	130	131	132	138	136
	°C	53.3	55.0	54.4	55.0	55.6	58.9	57.8
Gel-to-Peak Exo.	minutes	NA	5.22	NA	2.76	3.67	6.99	5.51
140°F (60.0°C)								
Gel Time	minutes	11.30	14.90	9.95	9.68	9.75	7.28	8.93
Cure Time	minutes	15.6	20.0	13.7	13.2	13.9	11.6	12.0
Peak Exotherm	°F	234	296	303	307	297	271	312
	°C	112	147	151	153	147	133	156
Gel-to-Peak Exo.	minutes	4.32	5.13	3.70	3.50	4.17	4.34	3.07
160°F (71.1°C)								
Gel Time	minutes	3.23	3.93	2.88	2.87	2.65	2.02	2.67
Cure Time	minutes	5.20	5.80	4.55	4.32	4.17	3.35	3.88
Peak Exotherm	°F	294	361	358	363	360	316	360
	°C	146	183	181	184	182	158	182
Gel-to-Peak Exo.	minutes	1.97	1.87	1.67	1.45	1.52	1.33	1.21
180°F (82.2°C)								
Gel Time	minutes	0.90	1.07	0.73	0.87	0.78	0.58	0.60
Cure Time	minutes	1.95	1.75	1.47	1.55	1.42	1.32	1.12
Peak Exotherm	°F	331	394	394	394	400	368	400
	°C	166	201	201	201	204	187	204
Gel-to-Peak Exo.	minutes	1.05	0.68	0.73	0.68	0.64	0.74	0.52
STABILITIES								
50°F (10.0°C)	days	33	20	19	19	20	13	16
65°F (18.3°C)	hours	113	84.5	81	67.5	78	66	54.5
77°F (25.0°C)	hours	44.5	33.6	30	30	30.5	27	25
90°F (32.2°C)	hours	13	10.5	14.5	10.5	8.5	10.5	8.5

DATA

Table 2: System II - Results using Di-(4-t-butylcyclohexyl)-peroxydicarbonate as the low temperature initiator and t-butyl peroxy-2-ethylhexanoate as the high temperature initiator.

Low Temp. Initiator, %		0.56	0.56	0.75	0.75	0.75	0.94	0.94
High Temp. Initiator, %		0.00	0.38	0.19	0.19	0.19	0.00	0.38
Test	Unit							
120°F (48.9°C)								
Gel Time	minutes	56.2	60.5	48.1	49.0	53.6	35.3	45.0
Cure Time	minutes	56.2	60.5	55.7	55.3	57.4	39.7	46.3
Peak Exotherm	°F	127	129	136	135	131	136	141
	°C	52.8	53.9	57.8	57.2	55.0	57.8	60.6
Gel-to-Peak Exo.	minutes	NA	NA	7.6	6.3	3.74	4.5	1.33
140°F (60.0°C)								
Gel Time	minutes	11.5	13.3	9.92	10.1	10.6	7.75	9.28
Cure Time	minutes	16.1	17.1	13.2	13.4	14.3	10.6	11.7
Peak Exotherm	°F	216	299	300	302	295	296	315
	°C	102	148	149	150	146	147	157
Gel-to-Peak Exo.	minutes	4.55	3.75	3.27	3.27	3.63	2.88	2.44
160°F (71.1°C)								
Gel Time	minutes	3.30	3.73	2.85	2.93	2.85	2.12	2.75
Cure Time	minutes	5.15	5.42	4.40	4.42	4.17	3.55	3.9
Peak Exotherm	°F	293	360	351	353	345	316	365
	°C	145	182	177	178	174	158	185
Gel-to-Peak Exo.	minutes	1.85	1.69	1.55	1.49	1.32	1.43	1.15
180°F (82.2°C)								
Gel Time	minutes	0.95	0.98	0.77	0.70	0.70	0.50	0.50
Cure Time	minutes	2.02	1.55	1.37	1.25	1.28	1.20	0.97
Peak Exotherm	°F	334	404	400	398	396	360	406
	°C	168	207	204	203	202	182	208
Gel-to-Peak Exo.	minutes	1.07	0.57	0.60	0.55	0.58	0.70	0.47
STABILITIES								
50°F (10.0°C)	days	32	12	12	16	18	17	11
65°F (18.3°C)	hours	120	70	68	65	66	71.5	48
77°F (25.0°C)	hours	44.5	28.5	26.5	27	29	28	21.5
90°F (32.2°C)	hours	14.5	10	9	9	10	9.5	8

DATA

Table 3: System III - Results using Di-(4-t-butylcyclohexyl)-peroxydicarbonate as the low temperature initiator of 25% 1,1-Di-(butylperoxy)-3,3,5-trimethylcyclohexanoate, 25% dibutyl phthalate and 50% t-butyl peroxy-2-ethylhexanoate as the high temperature initiator.

Low Temp. Initiator, %		0.56	0.56	0.75	0.75	0.75	0.94	0.94
High Temp. Initiator, %		0.00	0.38	0.19	0.19	0.19	0.00	0.38
Test	Unit							
120°F (48.9°C)								
Gel Time	minutes	62.6	67.8	52.0	51.3	51.3	38.8	35.1
Cure Time	minutes	62.6	67.8	57.1	54.4	54.0	38.8	37.9
Peak Exotherm	°F °C	125 51.7	128 53.3	132 55.6	131 55.0	131 55.0	130 54.4	132 55.6
Gel-to-Peak Exo.	minutes	NA	NA	5.13	3.15	2.73	NA	2.83
140°F (60.0°C)								
Gel Time	minutes	14.2	10.4	10.2	9.93	7.80	8.42	9.28
Cure Time	minutes	16.8	18.7	14.1	13.8	13.7	10.6	11.4
Peak Exotherm	°F °C	232 111	292 144	295 146	283 139	288 142	272 133	312 156
Gel-to-Peak Exo.	minutes	4.85	4.53	3.75	3.62	3.79	2.75	2.95
160°F (71.1°C)								
Gel Time	minutes	3.50	3.67	2.70	2.88	2.98	2.43	2.55
Cure Time	minutes	5.48	5.23	4.02	4.33	4.40	3.80	4.25
Peak Exotherm	°F °C	289 143	349 176	342 172	352 178	345 174	313 156	362 183
Gel-to-Peak Exo.	minutes	1.98	1.56	1.32	1.45	1.42	1.37	1.70
180°F (82.2°C)								
Gel Time	minutes	1.00	0.95	0.72	0.83	0.73	0.57	0.58
Cure Time	minutes	2.00	1.62	1.32	1.37	1.28	1.27	1.15
Peak Exotherm	°F °C	326 163	393 201	392 200	381 194	388 198	357 181	387 197
Gel-to-Peak Exo.	minutes	1.00	0.67	0.60	0.53	0.55	0.70	0.57
STABILITIES								
50°F (10.0°C)	days	38	19	18	22	20	21	17
65°F (18.3°C)	hours	122	91	64.5	78	84.5	78.5	66
77°F (25.0°C)	hours	45.5	35.5	31.5	32	32	27.5	24.5
90°F (32.2°C)	hours	14	12.5	10	10	10	9	8.5

DATA

Table 4: System IV - Results using Di-(4-t-butylcyclohexyl)-peroxydicarbonate as the low temperature initiator and t-butyl perxy-3,5,5-trimethyl hexanoate as the high temperature initiator.

Low Temp. Initiator, %		0.56	0.56	0.75	0.75	0.75	0.94	0.94
High Temp. Initiator %		0.00	0.38	0.19	0.19	0.19	0.00	0.38
Test	Unit							
120°F (48.9°C)								
Gel Time	minutes	60.0	75.5	50.4	52.9	55.3	33.8	41.7
Cure Time	minutes	60.0	75.5	50.4	52.9	55.3	39.3	48.1
Peak Exotherm	°F	125	125	128	127	128	136	138
	°C	51.7	51.7	53.3	52.8	53.3	57.8	58.9
Gel-to-Peak Exo.	minutes	NA	NA	NA	NA	NA	5.4	6.33
140°F (60.0°C)								
Gel Time	minutes	11.3	14.9	10.2	9.95	9.83	7.43	8.55
Cure Time	minutes	16.3	20.0	14.1	14.1	13.7	10.6	11.8
Peak Exotherm	°F	225	267	300	289	300	269	319
	°C	107	131	149	143	149	132	159
Gel-to-Peak Exo.	minutes	5.03	5.08	3.88	4.13	3.87	3.17	3.25
160°F (71.1°C)								
Gel Time	minutes	3.33	3.27	2.92	2.90	2.73	2.28	2.57
Cure Time	minutes	5.22	5.33	4.45	4.37	4.22	3.73	3.88
Peak Exotherm	°F	290	348	351	354	355	319	368
	°C	143	176	177	179	179	159	187
Gel-to-Peak Exo.	minutes	1.89	2.06	1.53	1.47	1.49	1.45	1.31
180°F (82.2°C)								
Gel Time	minutes	0.97	1.12	0.77	0.77	0.77	0.58	0.67
Cure Time	minutes	2.02	1.82	1.42	1.37	1.43	1.30	1.20
Peak Exotherm	°F	335	388	388	397	388	363	401
	°C	168	198	198	203	198	184	205
Gel-to-Peak Exo.	minutes	1.05	0.70	0.65	0.60	0.66	0.72	0.53
Stabilities								
50°F (10.0°C)	days	36	27	24	21	22	18	17
65°F (18.3°C)	hours	129	111	83	93	88	80	63.5
77°F (25.0°C)	hours	43	41.5	31	30.5	31	25	24
90°F (32.2°C)	hours	14	13.5	11	10.5	11	9	9

Table 5: Least Squares Analysis of Gel Time/Stability Data

System I	Run	Slope	Intercept	R²
	1	-0.0359	6.2226	0.9924
	2	-0.0334	5.9227	0.9951
	3	-0.0349	6.0231	0.9955
	4	-0.0342	5.9211	0.9929
	5	-0.0347	5.9641	0.9930
	6	-0.0351	5.9098	0.9948
	7	-0.0343	5.8513	0.9944
	Average	-0.0347	5.9735	0.9940
	Maximum	-0.0334	6.2226	0.9955
	Minimum	-0.0359	5.8513	0.9924
	Standard Deviation	0.00078	0.1217	0.0012
System II	Run	Slope	Intercept	R²
	1	-0.0358	6.2263	0.9923
	2	-0.0325	5.7520	0.9979
	3	-0.0335	5.7937	0.9970
	4	-0.0341	5.8693	0.9956
	5	-0.0344	5.9272	0.9956
	6	-0.0358	6.0027	0.9954
	7	-0.0336	5.7326	0.9975
	Average	-0.0342	5.9005	0.9959
	Maximum	-0.0325	6.2263	0.9979
	Minimum	-0.0358	5.7326	0.9923
	Standard Deviation	0.0012	0.1732	0.0019
System III	Run	Slope	Intercept	R²
	1	-0.0359	6.2573	0.9909
	2	-0.0340	5.9808	0.9971
	3	-0.0345	5.9368	0.9953
	4	-0.0347	5.9954	0.9929
	5	-0.0349	6.0061	0.9948
	6	-0.0357	6.0299	0.9930
	7	-0.0349	5.9078	0.9934
	Average	-0.0349	6.0163	0.9939
	Maximum	-0.0340	6.2573	0.9971
	Minimum	-0.0359	5.9078	0.9909
	Standard Deviation	0.0007	0.1141	0.0020
System IV	Run	Slope	Intercept	R²
	1	-0.0360	6.2573	0.9912
	2	-0.0348	6.1230	0.9946
	3	-0.0351	6.0445	0.9930
	4	-0.0350	6.0294	0.9944
	5	-0.0351	6.0454	0.9944
	6	-0.0355	5.9836	0.9929
	7	-0.0345	5.8833	0.9936
	Average	-0.0352	6.0524	0.9934
	Maximum	-0.0345	6.2573	0.9946
	Minimum	-0.0360	5.8833	0.9912
	Standard Deviation	0.0005	0.1161	0.0012

Table 6: Least Squares Analysis of Cure Time/Stability Data

System I	Run	Slope	Intercept	R²
	1	-0.0247	4.7069	0.9946
	2	-0.0267	5.0426	0.9999
	3	-0.0258	4.7857	0.9975
	4	-0.0253	4.7048	0.9955
	5	-0.0260	4.8085	0.9981
	6	-0.0248	4.5439	0.9960
	7	-0.0269	4.8765	0.9986
	Average	-0.0257	4.7813	0.9972
	Maximum	-0.0247	5.0426	0.9999
	Minimum	-0.0269	4.5439	0.9946
	Standard Deviation	0.0009	0.1559	0.0019
System II	Run	Slope	Intercept	R²
	1	-0.0358	6.2263	0.9923
	2	-0.0325	5.7520	0.9979
	3	-0.0335	5.7937	0.997
	4	-0.0341	5.8693	0.9956
	5	-0.0344	5.9272	0.9956
	6	-0.0358	6.0027	0.9954
	7	-0.0336	5.7326	0.9975
	Average	-0.0342	5.9005	0.9959
	Maximum	-0.0325	6.2263	0.9979
	Minimum	-0.0358	5.7326	0.9923
	Standard Deviation	0.0012	0.1732	0.0019
System III	Run	Slope	Intercept	R²
	1	-0.0249	4.7449	0.9964
	2	-0.0271	5.0708	0.9995
	3	-0.0273	4.9975	0.9974
	4	-0.0265	4.8862	0.9980
	5	-0.0268	4.9322	0.9987
	6	-0.0245	4.4973	0.9973
	7	-0.0249	4.5676	0.9974
	Average	-0.0260	4.8138	0.9978
	Maximum	-0.0245	5.0708	0.9995
	Minimum	-0.0273	4.4973	0.9964
	Standard Deviation	0.0012	0.2178	0.0010
System IV	Run	Slope	Intercept	R²
	1	-0.0246	4.6877	0.9951
	2	-0.0271	5.1106	0.9976
	3	-0.0258	4.7759	0.9993
	4	-0.0263	4.8629	0.9990
	5	-0.0264	4.8708	0.9966
	6	-0.0245	4.4962	0.9969
	7	-0.0265	4.8233	0.9974
	Average	-0.0259	4.8039	0.9974
	Maximum	-0.0245	5.1106	0.9993
	Minimum	-0.0271	4.4962	0.9951
	Standard Deviation	0.0010	0.1877	0.0014

FIGURE 1: System III - Gel Time

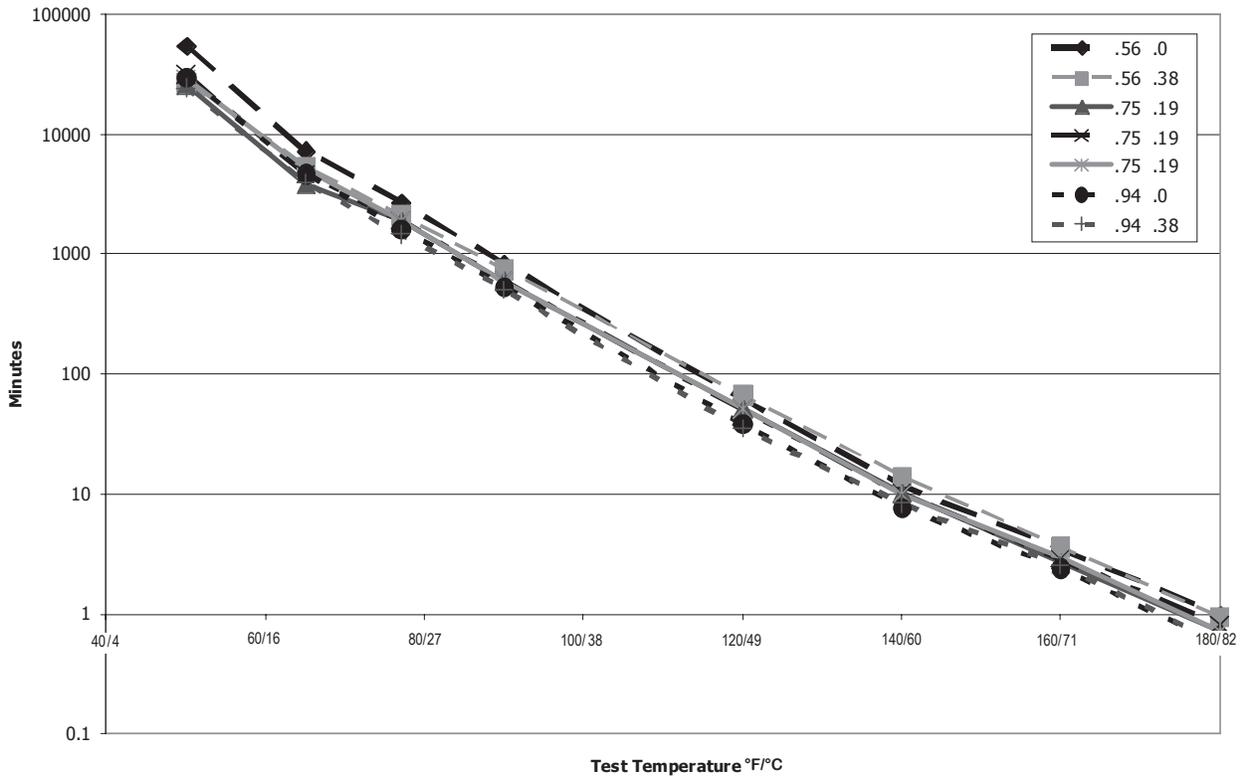


FIGURE 2: System III - Cure Time

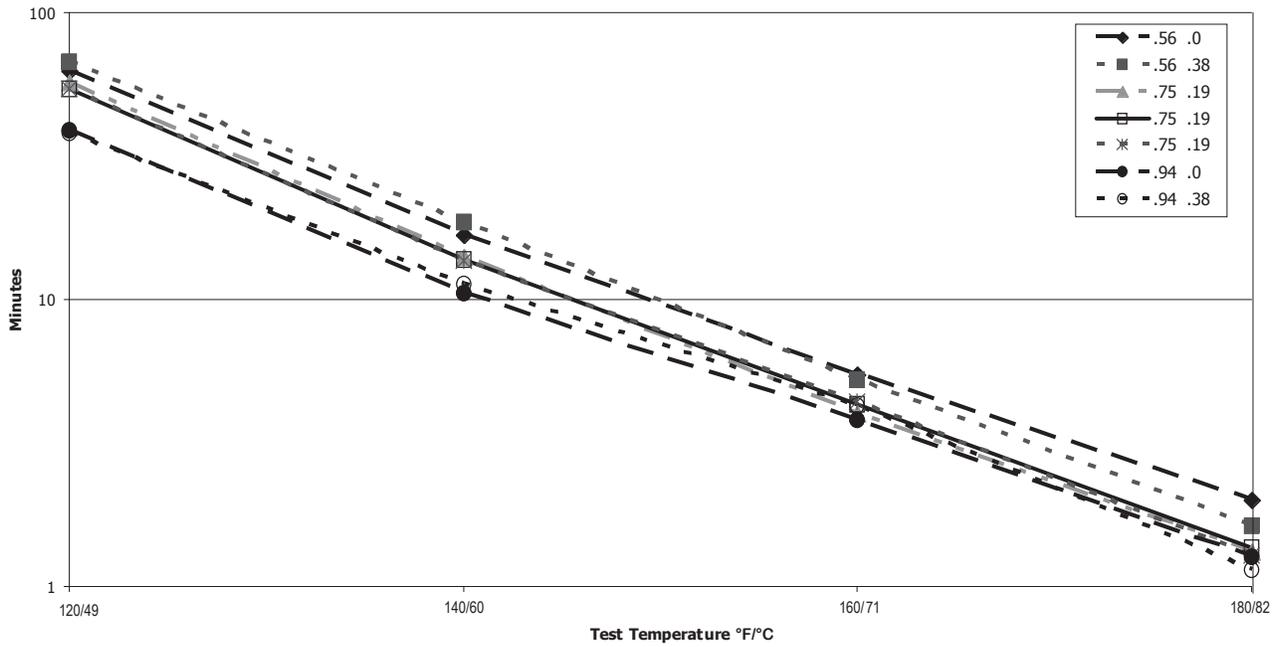


FIGURE 3: System III Gel-to-Peak Time

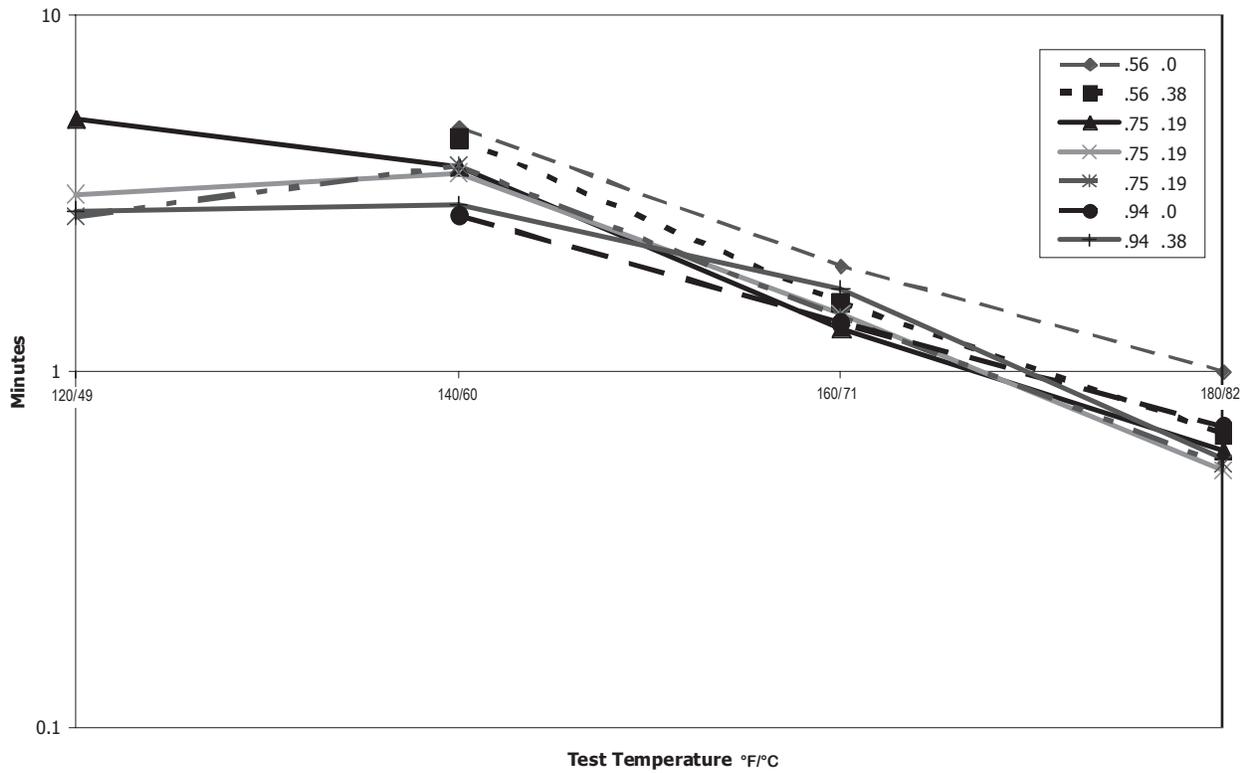
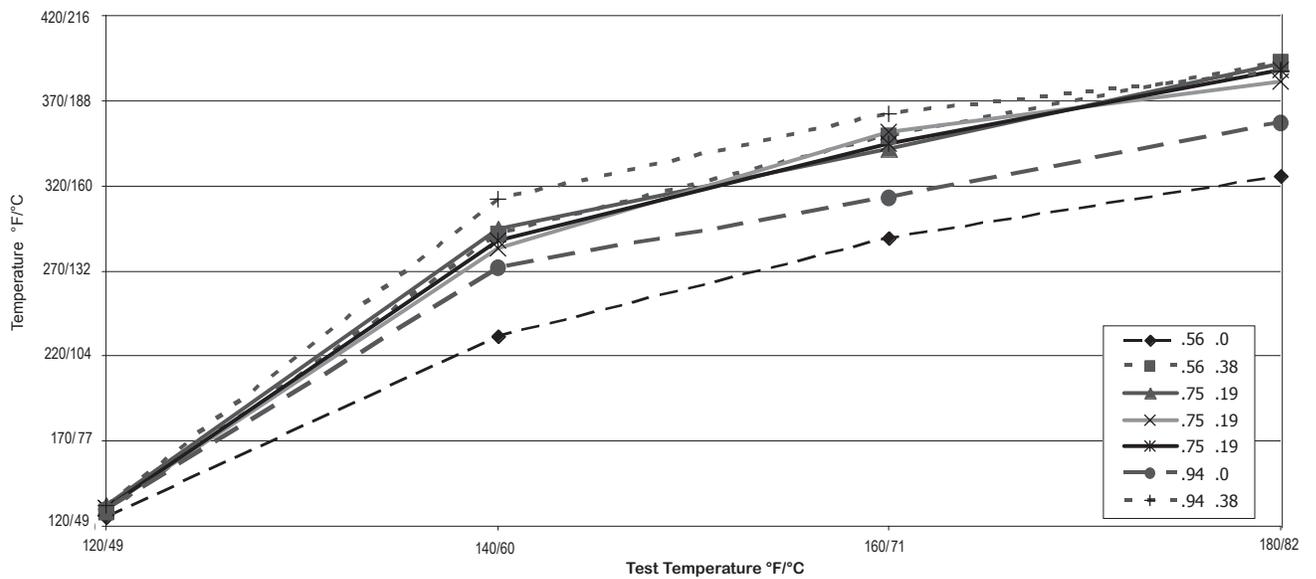
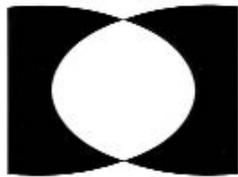


FIGURE 4: System III Peak Exotherm





INTERPLASTIC CORPORATION
Thermoset Resins Division

1225 Willow Lake Blvd., St. Paul MN 55110-5145
800.736.5497 Fax: 651.481.9836
www.interplastic.com